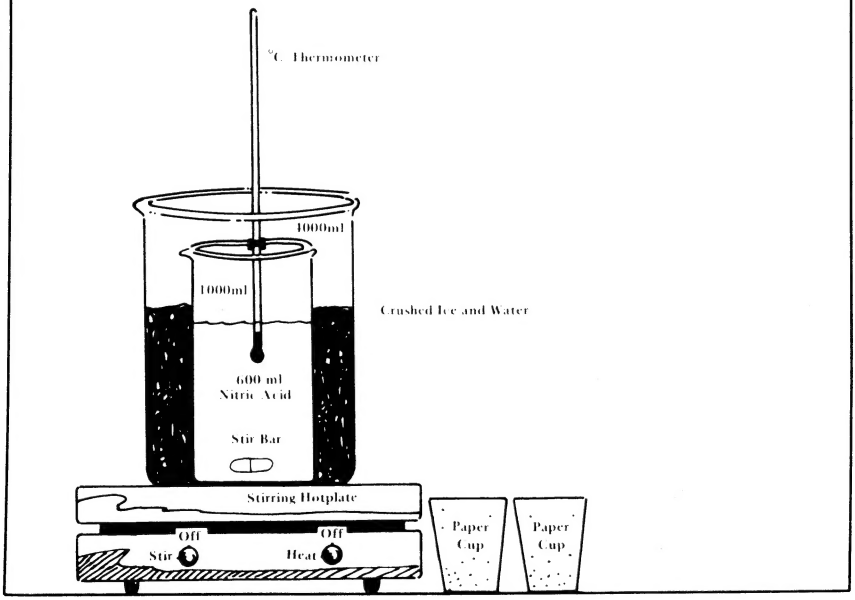
THE BIG BANG

IMPROVISED PETN & MERCURY FULMINATE



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The Big Bang: Improvised PETN and Mercury Fulminate
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WARNING

There may be certain federal, state or local laws which prohibit the possession or manufacture of certain substances mentioned herein. Severe penalties may be prescribed for violation of such laws. Be warned!

The procedures to be followed in this manual and the resulting end-product are extremely dangerous. Whenever dealing with high explosives, special precautions should be followed in accordance with industry standards for experimentation and production of high explosives. Failure to strictly follow such industry standards may result in harm to life or limb.

Therefore, the author and publisher disclaim any liability from any damages or injuries of any type that a reader or user of information contained within this manual may encounter from the use of said information. Use this manual and any end-product or by-product at your own risk.

Preface

The information in this manual serves as an introduction to the organic explosive PETN, pentaerythritol tetranitrate. Included are important physical and chemical properties with which the reader should be familiar before proceeding to the section that describes in detail one of several proven methods for the manufacture of high-grade PETN. Also included is a section detailing the process for making mercury fulminate, an excellent initiating agent for detonating PETN or any other explosive material. Finally, it shoud be mentioned that the chemicals listed in this text are hazardous if used improperly or handled carelessly. The reader is urged to adopt a "think before you do" attitude, pay attention to details, and use safe laboratory equipment and procedures.

1. PETN: Its Discovery and Properties

The high explosive pentaerythritol tetranitrate was first synthesized in 1894 at the Rheinish-Westfalische Sprengstoff Company in Germany by a chemist researching the nitration process of pentaerythritol in a high concentration of nitric acid. The resulting explosive was collected, tested, and found to have a very fast rate of detonation, high density, and good chemical stability, making it an ideal substance for military use. Unfortunately, at that time there were no means available to mass-produce the raw ingredient pentaerythritol. PETN thus remained a laboratory interest until World War II, when it was used in ammunitoin, bombs, and other fragmentation devices. During this time, Germany produced as much as 1,440 tons of PETN per month, with the USA and the USSR following suit.

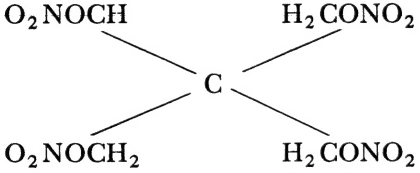
Still manufactured on a large scale, PETN remains one of the most powerful conventional explosives ever developed, rivaled only by HMX and RDX. The military, as might be expected, is the largest user of PETN, with annual purchasing well over 2,000 tons. Non-military use is primarily limited to demolition work and as booster material for secondary explosives such as ANFO, dynamite, etc.

PHYSICAL AND CHEMICAL PROPERTIES

PETN is a white crystalline substance that feels powdery to the touch. In its pure form, PETN melts at 141.3 degrees centigrade. The boiling point is:

160°C at 2mm Hg
180°C at 50mm Hg
200°C at 760mm Hg (standard atmosphere)

The molecular structure is:



$C_5H_8O_{12}N_4$

The calculated nitrogen content is as follows:

Element	**# Atoms x Atomic Wt.**		
Carbon	5 x 12.011	=	60.055
Hydrogen	8 x 1.00794	=	8.064
Oxygen	12 x 15.9994	=	191.993
Nitrogen	4 x 14.0067	=	56.027
	Total		316.139

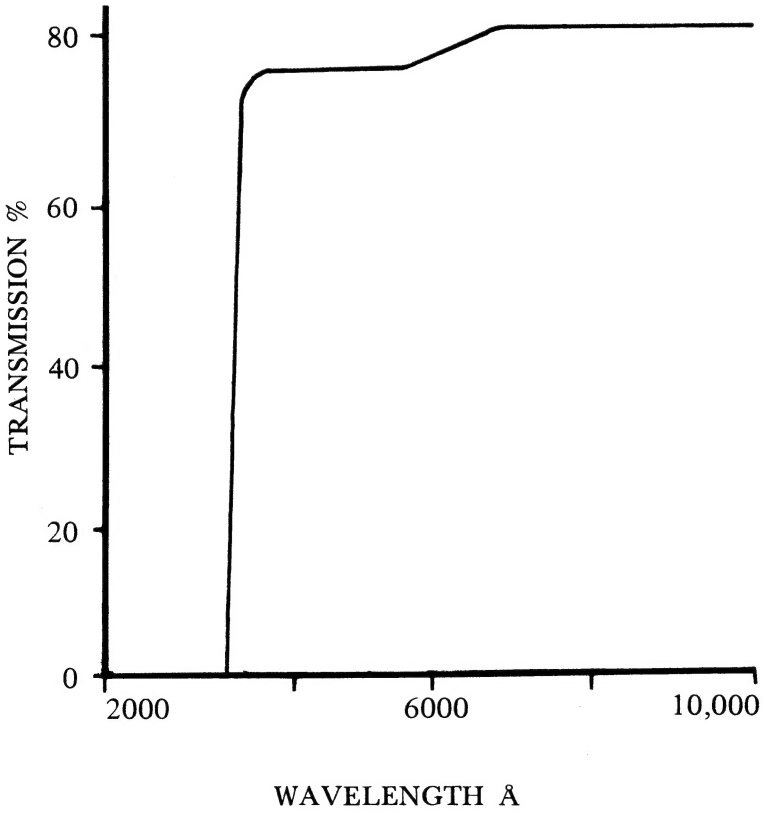
$$\frac{56.027}{316.139}\,(100) = 17.722\%\ \text{Nitrogen}$$

Correspond with the military specification MIL-P-387 for PETN:

Melting Point	140.0°C

Nitrogen Content	17.5%	Minimum
Substances Insoluble in Acetone	.10%	Maximum
Acid or Alkali Content	.003%	Maximum
Vacuum Stability at 120°C	5 ml of gas/ 20 hours	
Overall Purity	98.7%	

If we examine the ultraviolet absorption spectrum of a single PETN crystal, we find a narrow band in the 280μm area. Because ordinary sunlight emits UV in the same range, PETN should not be exposed to the sun for a long period of time as damage in the form of cracks in the crystals will appear and render the PETN useless.



2. PETN Manufacture

PETN can be made in a number of different ways, but the overall process is always the same and consists of the following steps:

1. Nitration. Adding pentaerythritol to a given amount of nitric acid for a specific time and at a specific temperature to form crude PETN.

2. Washing. The crude PETN is rinsed with distilled water until free of acid, filtered, and thoroughly dried.

3. Recrystallization. The dry PETN is dissolved in heated acetone, purified with a neutralizing agent, and then poured into cold water where the purified PETN is precipitated.

4. Washing. The pure PETN is again rinsed with distilled water to remove remaining traces of acid and acetone, filtered, and dried to a 10 percent water content. The PETN is now ready for use.

We will now proceed to the actual method for making high-grade PETN on a laboratory scale (larger amounts can be made by increasing the size of the equipment) that will yield about 500ml per batch. The amount of chemicals used can vary if one wishes to produce more or less PETN per batch. However, the proportions and tem-

peratures are exact and must not be changed under any circumstances. We will begin with a list of equipment and chemicals needed. Then each of the steps mentioned above will be detailed and fully discussed.

HARDWARE REQUIRED

1. Stirring hotplate, with at least a 10" x 10" surface area. This type of hotplate will heat solutions and stir them by spinning a glass-coated bar magnet that is dropped into the solution being treated. The temperature and mixing rate are both adjustable by turning two dials on the face of the machine. The magnetic stir bar should be about 2.75 inches long and glass coated. If a regular hotplate of the nonstirring type is used, then all stirring must be done by hand with a glass rod.
2. Glass-coated magnetic stir bar 2.75" long
3. One 4,000ml Pyrex® beaker of standard configuration, with graduation
4. One 1,000ml Pyrex® beaker, tall form with graduation
5. One thermometer graduated in degrees centigrade
6. One thermometer clip to hold thermometer to the beaker
7. A few glass stirring rods
8. Pack of fine-grade filter paper (grade: 230, 33cm diameter)
9. Glass funnel
10. Plastic bucket: one or two
11. Wood or plastic cookspoon (used to scoop PETN crystals)
12. Several pans for drying (disposable pizza pans work well)
13. One graduated cylinder used for measurement. A 100 or 200ml cylinder will do.
14. Several paper cups
15. Full face shield to protect eyes and face while working with acid

16. Plastic or rubber gloves

CHEMICALS REQUIRED

The amount of chemicals listed is just enough to make one 500ml lot of PETN. The reader will have to decide how much to purchase if he decides to vary the amount of PETN produced.

1. 600ml nitric acid with a purity of 98 percent. (Don't use 70 percent or 90 percent—use only 98 percent.)
2. 250ml pentaerythritol, reagent grade
3. 1,500ml acetone, reagent grade
4. One teaspoon sodium carbonate powder, reagent grade
5. 800ml distilled water
6. 30 to 40 gallons of tap water for rinsing. Should tap water be hard or contain chlorine, distilled water must be used.
7. One vial of red litmus paper, used to test pH level alkaline range 7 to 14
8. One vial of blue litmus paper, used to test pH level in acid range 0 to 7
9. Small bag of crushed ice
10. Optional: Full range 0 to 14 pH indicator strips. These are helpful in determining the pH of a solution, but litmus paper will suffice.

All of the hardware and chemicals listed are readily available from major scientific supply companies. Consult the yellow pages and don't be afraid to shop around for the best prices.

PROCEDURE

Step One: Nitration

(a) Take the 1000ml tall form beaker and fill it with

600ml of nitric acid. Be sure to wear your face shield when working with acid.

(b) Place the tall form beaker with the acid inside of the larger 4000ml beaker and center it.

(c) Fill the larger beaker with crushed ice up to the same level as the acid in the tall form beaker. Now pour tap water over the ice until the level reaches the 700ml mark on the tall form beaker. *Do not let any ice or water fall into the acid.*

(d) Carefully place the stir bar into the beaker of acid.

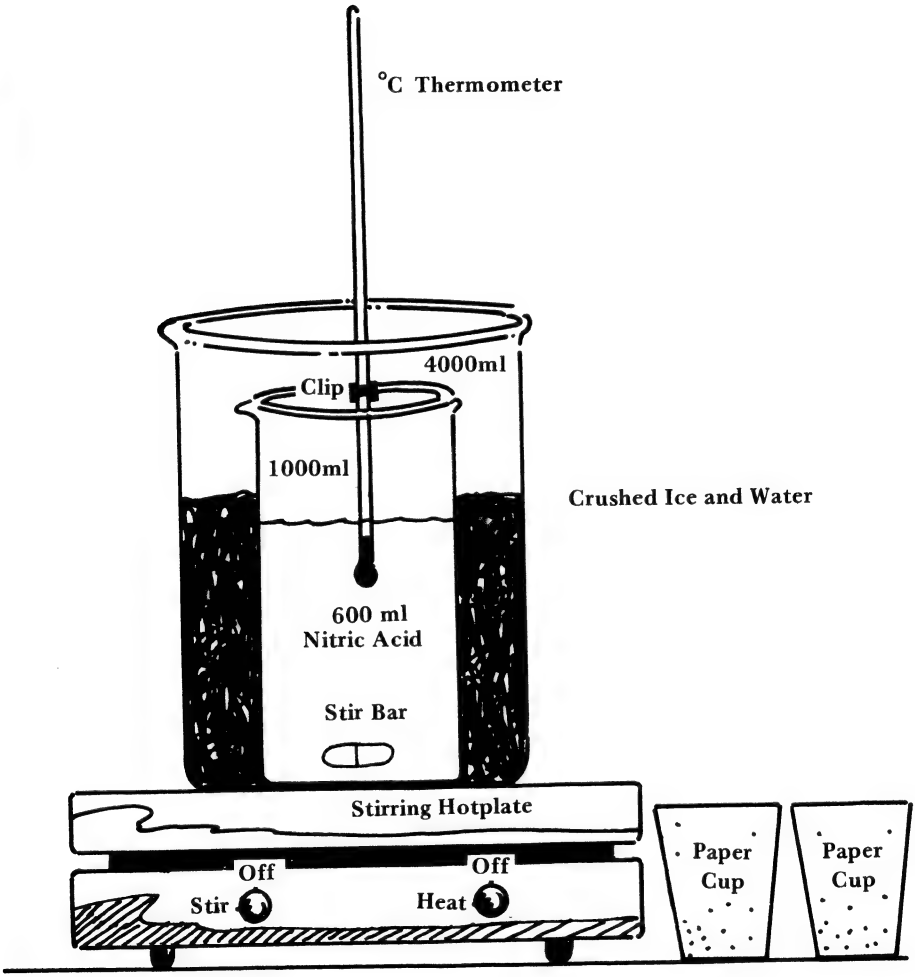
(e) Using the thermometer clip, attach the thermometer to the rim of the tall form beaker and adjust it so that the bulb (the part with the mercury) falls just slightly below the surface of the acid. Be sure the numbers can be clearly read.

(f) With two hands, carefully lift the whole assembly and place it on the stirring hotplate and move it around so that the stir bar is centered inside the tall form beaker. All controls on the hotplate should be in the off position.

(g) Using the graduated cylinder, measure out 250ml of pentaerythritol and pour this into the paper cups (you will most likely need two paper cups).

This assembly is called a nitrator, and should look like the diagram on page 9.

NITRATOR


°C Thermometer
4000ml
Clip
1000ml
Crushed Ice and Water
600 ml
Nitric Acid
Stir Bar
Stirring Hotplate
Off
Stir
Off
Heat
Paper
Cup
Paper
Cup
250ml
Pentaerythritol

During the manufacture process, acetone is used to purify crude PETN and to safely remove crystals that may have condensed on various pieces of laboratory equipment. Because acetone readily dissolves PETN, a table of solubility is given below as a reference.

Amount of PETN dissolved in 100 grams of solution (grams)	ACETONE CONCENTRATION IN WATER				
	55%	70%	80%	90%	92%
	TEMPERATURE OF SOLUTION IN °C				
5	1 gr @ 41C	41.5	22		
10	4 gr @ 62	54.5	38.5	15	10
15		62	48	24.5	20.5
20			54	34.5	29
25			59	41.5	34
30			63	46.5	40.5
35				51.5	45
40				55	50
45				58.5	54
50				61.5	57.5
55					60.5
60					62.5

The above table indicates a low solubility of PETN in water and this helps to explain the low toxicity of PETN on the human body. Handling or inhaling the dust in small amounts causes *almost* no effect. From a toxic point of view, PETN, unlike nitroglycerine, TNT, or dynamite, is safe to work with.

Explosive Properties

Pure PETN, when heated above its melting point, explodes at 205-225°C.

A TABLE OF GASEOUS PRODUCTS RELEASED DURING DECOMPOSITION OF PETN

INITIATION	NO_2	NO	N_2O	N_2	CO_2	CO	H	O_2
Detonation	–	5.3	–	22.8	37.0	26.7	6.8	1.4
Impact	–	24.3	5.3	9.4	19.1	35.4	6.5	–
Thermal 210°C	12.0	47.6	9.5	1.6	6.3	21.0	2.0	–

Heat of Explosion: 1,530 Kcal/Kg.
Volume of Gases: 768 L/Kg.
Temperature: 4,230°C

The rate of detonation of a single crystal of PETN, the density of which is 1.77g/cubic centimeter, is 8,500 meters/second. In order to get a cartridge filled with PETN powder to detonate at the same rate, two things must be done:

1. Pure, dry PETN Must be mixed by weight with 10 percent water. This will drive the air out and allow the detonation wave to travel rapidly from one crystal to the next. The water also makes the PETN safer to work with, as dry PETN explodes when subjected to a slight impact.
2. The PETN must be loaded into the cartridge under high pressure so that the density of the powder approaches the crystal density.

The ideal loading density of a cartridge containing PETN should be in the range of 1.72 to 1.73g/cubic centimeter. At this density, the 8,500 meter/second detonation rate can be achieved and the maximum explosive power of PETN obtained. It should be mentioned, however, that even if a cartridge is only packed by hand, a very powerful explosive device will still result.

The loading density of a cylindrical cartridge can be determined by using the following equations:

$$\text{Pressure} = \frac{\text{Force}}{\text{Area}}$$

$$\text{Volume} = \text{Area} \times \text{Length}$$

$$\text{Loading Density} = \frac{\text{Mass (Amount of PETN)}}{\text{Volume of Cartridge}}$$

We are now ready to continue. Be sure the work area has good ventilation, and wear your face shield to

avoid acid burns in case any beakers are accidentally knocked over.

(h) Turn on the stirrer and adjust until a smooth rapid motion occurs in the acid. The heater will be in the "off" position during the whole nitration process. If you are not using a stir bar, the stirring must be continuously done by hand with a glass rod.

(i) Look at the thermometer. When the temperature falls to 10°C, very slowly sprinkle in some of the pentaerythritol by squeezing the rim of the cup to form a spout and shake lightly. As soon as the temperature starts to rise, stop adding. Look to see where the temperature stops. If it is below 20°C, then continue adding the pentaerythritol and stopping whenever the thermometer reads 20°C. Allow the whole mixture to cool back down to 10°C and then start sprinkling in the pentaerythritol until 20°is again obtained. Continue adding in this manner until all 250ml of pentaerythritol has been added. When the temperature again falls to 10°C, remove the thermometer and the stir bar. Take the small beaker out of the larger one and set it aside for forty-five minutes. The stir switch can be turned off and the ice-water mixture thrown out.

Important: Never let the temperature of the mixture rise above 25°C during this operation.

This concludes Step One.

Step Two: First Washing

(a) After forty-five minutes have elapsed, the crude PETN will have settled to the bottom of the beaker as a thick yellow-white sludge, with a layer of acid on top. Pick up the beaker and slowly pour off the top layer of acid, being careful

not to disturb the PETN sludge. When finished, set the beaker aside and dispose of the waste acid.

(b) Fill a plastic bucket with two gallons of water. Use distilled water if your tap water is hard or chemically treated.

(c) Pour the beaker of PETN into the bucket and scoop out any sludge with a wood cook spoon. Using a glass rod, stir the contents of the bucket vigorously for ten seconds and then wait for the PETN to settle. If a foam has developed on the surface of the water, be sure to agitate the surface with your stirring rod. The foam, which is just air trapped in PETN, will settle to the bottom of the bucket.

(d) After the contents have settled, take a strip of blue litmus paper and dip it into the bucket. You will notice that the litmus paper turns red, indicating the presence of acid. This acid must be removed, so take the bucket and slowly pour off the layer of water. Be careful not to pour off any PETN by accident.

(e) Take two gallons of fresh water and pour it into the bucket. As before, stir to break up the foam and then allow the contents to settle again. Using a new blue litmus paper, test for the presence of any acid. Should acid be present, continue the cycle of pouring off the water adding fresh water, stirring, settling, and testing with litmus paper.

(f) After several wash cycles, you will find that the litmus paper no longer turns red but instead remains blue, indicating that the rinse water is now neutral. Wash the PETN one more time, let settle, and pour off the water.

(g) Place a filter paper into the glass funnel. Scoop the PETN from the bottom of the bucket and put it into the filter. The filter paper and funnel serve to allow the water to drip away, thus allowing the PETN to dry faster. Allow the PETN to filter for at least thirty minutes.

(h) After filtering, take the PETN and spread it out as a thin layer in the drying pans. The PETN must be allowed to dry completely. When dry, it will be a white crystalline powder. The drying might take several days.
(i) Clean and dry the beakers, stir bar, and thermometer, as they will be used again during the next step.
(j) After the PETN has dried, place it in any handy container—other than the beakers—and set it aside.

This concludes Step Two.

Step Three: Recrystallization

(a) Take one teaspoon of sodium carbonate and place it in the tall form beaker. Add 800ml of distilled water and stir until completely dissolved. This, your neutralizing solution, will be used shortly.
(b) Fill the 4,000ml beaker with 1,500ml of acetone. Put in the stir bar and thermometer and place on top of the hotplate. Turn the stir dial to a medium speed and turn on the heat. The temperature of the acetone will rise and *must* be held between 50°C and 60°C. *Do not* let the temperature stray out of this range during the next steps. Aim for a temperature of 55°C. Heated acetone is *highly flammable*—use caution!
(c) Take two heaping tablespoons of dry PETN, add it to the acetone, and let it dissolve completely. The temperature will now drop slightly, but if it is still above 50°C, take two more tablespoons of PETN, add it to the acetone, and let it dissolve. Keep up this method of adding two tablespoons of PETN and letting it dissolve until the temperature falls to 50°C. When the temperature reaches 50°C, stop adding PETN and let the temperature rise to just under 60°C. Then begin adding PETN again two spoons at a time and allowing it to dissolve until all the PETN

has dissolved.

(d) With all the PETN now dissolved in acetone and the temperature held at 55°C, we will begin neutralizing the solution.

(e) Have both the blue and red litmus papers ready.

(f) Fill the graduated cylinder with the sodium carbonate solution that was prepared in the tall form beaker.

(g) Keeping the contents of the large beaker in the 50° to 60°C range, add to it 10ml of the sodium carbonate solution (SCS). Insert a blue litmus paper. If the paper turns red, add 10ml more of the SCS, and test again with the litmus paper. Keep up this process of adding 10ml of SCS at a time, until the litmus paper remains blue. When this happens, add a final 10ml of SCS. Test with both the red and blue litmus paper. If neither paper changes color, the solution is neutral. Turn off the hotplate.

(h) Immediately fill a bucket with two gallons of cold water. Pour the contents of the large beaker into the bucket and stir. The PETN will now precipitate with its new fine crystalline structure.

(i) Test the water in the bucket with blue and red litmus paper. If the instructions in this manual were followed carefully, there will be no reactions. If they do react, then an error was made and salvaging the PETN will be beyond the scope of this manual. You will have to try again.

This concludes Step Three.

Step Four: Second Washing

(a) This washing process is similar to Step Two, only we will be removing acetone instead of acid from the PETN. When the contents in the bucket (including the foam) have settled, pour off the water.

(b) Add two gallons of fresh water, stir, and allow to settle. If you smell the water, you will notice a

strong acetone odor. Continue the wash cycle until the acetone odor is no longer apparent and then rinse the PETN two more times to complete the washing.

(c) After the PETN has settled, pour off the water. Filter the PETN and allow to dry to a 5 to 10 percent moisture content. The finished PETN should not feel wet when a sample is squeezed between your fingers. When the PETN has been dried, it will be ready for use. Store in a cool, dark place.

This concludes the manufacture of PETN.

3. Mercury Fulminate

PETN will not detonate if lit with a match or fuse, and so some kind of initiator is needed in the form of a blasting cap. Mercury fulminate makes a good filler for caps and is also very easy to make.

Before the process is detailed, the reader is advised that mercury fulminate is *very sensitive to heat, friction, impact, and static electricity.* When working with this substance, use small amounts at a time and wear eye protection.

HARDWARE NEEDED

1. Graduated cylinder
2. Two glass containers each with a minimum of 1,000ml capacity
3. One large, heat-resistant flask of one-gallon capacity. A large wine jug may work if a heat resistant flask is unavailable.
4. Glass funnel
5. Filter paper (same type as used for PETN)
6. Blue litmus paper

CHEMICALS NEEDED

1. 600ml 70 percent nitric acid, reagent grade
2. 160ml distilled water
3. 900ml ethanol alcohol, reagent grade
4. 8ml of mercury, reagent grade

Warning: Make no substitutions for these chemicals or an explosion may result.

PROCEDURE

(a) Pour 150ml distilled water into one of the glass containers. Add the 600ml of acid. Now add the 8ml of mercury to the acid and let dissolve *completely*. You will soon have a dark-green liquid. Allow this solution to cool to room temperature (65° to 70°F).

(b) Pour the 900ml of alcohol into the second container; temperature should be 65° to 70°F.

(c) Take the heat-resistant flask and both containers outdoors in the open and away from people, as the next step causes the release of toxic gases. *Do not do indoors.*

(d) Pour the container of alcohol into the flask first. Then add the green mercury acid solution to the flask. Use the funnel for easy pouring.

(e) Move away from the flask. In about ten to forty-five minutes, a thick white smoke will rise up out of the flask, indicating that the proper reaction is taking place.

(f) After allowing the flask to stand for three hours, fill it with distilled water. You will see the mercury fulminate crystals on the bottom of the flask.

(g) Pour the entire contents of the flask into a filter and wash the crystals with distilled water until the blue litmus paper indicates the water is neutral and free of acid.

(h) Remove the crystals from the filter and allow to

dry completely; store in a cool, dry place. The mercury fulminate is now ready for use. The material should be a gray-brown crystalline powder.

Warning: When filling blasting caps with mercury fulminate, do not use copper, brass, or aluminum jackets, as these metals react with mercury fulminate. Instead, use stainless steel, glass, paper, etc. The jackets should be about 2.75 to 3.00 inches long, and 0.25 inches in diameter. The loading density can be around two to three times the density of the loose material. The caps can be fired with a fuse or by heating wire.

4. Brisance

The term *brisance,* also known as *shattering effect,* is used to describe an explosive in relation to its ability to demolish a solid object, and is commonly used to compare different explosives. When an explosive detonates, tremendous pressure is released almost instantaneously in a shock wave that lasts for only a fraction of a second. The subsequent expansion of the gases performs work, but it is the sudden pressure that is created that will shatter, rather than displace, an object. Brisance is important because it tells a person the effectiveness of an explosive in fragmenting various devices. Brisance is approximately proportional to detonation velocity, force, and density loading. Some common figures follow.

EXPLOSIVE	BRISANCE %TNT	DENSITY (g/cc)	DETON. (m/sec)	POWER % TNT	GRAMS SAND CRUSHED
Black powder	22	1.6	400	10	
RDX	125-140	1.7	8049	150-170	53.2
Dynamite	–	–	5200	102.5	
HMX	125-155	1.84	9124	159-165	
Mercury ful.	49-55	4.42	5400	51-54	22.5
Nitroglycerin	120	1.6	7700	185	
PETN	129-141	1.7	8400	161-181	57.9
TNT	100	1.59	6970	100	41.42

As the above figures show, PETN has the highest ability to crush sand, and is the second-highest-rated explosive in terms of velocity. The other two most commonly used explosives, RDX and HMX, are much more difficult to make than PETN.